



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/AU96/00354 (22) International Filing Date: 13 June 1996 (13.06.96) (30) Priority Data: PN 3520 13 June 1995 (13.06.95) AU (71) Applicants (for all designated States except US): ICI AUSTRALIA OPERATIONS PTY. LTD. [AU/AU]; 1 Nicholson Street, Melbourne, VIC 3000 (AU). QUEENSLAND METALS CORPORATION LIMITED [AU/AU]; Level 6, 9 Sherwood Road, Toowong, QLD 4066 (AU). (72) Inventors; and (75) Inventors/Applicants (for US only): VINCENT, Adam, Hayden [AU/AU]; Unit 3, 5 Myrtle Grove, Blackburn, VIC 3130 (AU). McLEAN, Ian, Alexander [AU/AU]; 7 Sandy Creek Road, Riddells Creek, VIC 3431 (AU). (74) Agent: KELLY, Robin, Thomas; Fisher Adams Kelly, Level 1/349 Coronation Drive, P.O. Box 1477, Milton, QLD 4064 (AU).		(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>With amended claims.</i>
(54) Title: PEROXIDE BLEACHING OF PULP (57) Abstract <p>A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g. By using such parameters, a peroxide bleaching process may be carried out most efficiently on a commercial scale.</p>		

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GA	Gabon			VN	Viet Nam

TITLE

"PEROXIDE BLEACHING OF PULP"

FIELD OF THE INVENTION

5 THIS INVENTION relates to a process for peroxide bleaching of pulp. Pulps which may be bleached in the process of the invention include lignocellulose pulp which may be produced mechanically and chemi-mechanically with yields in the region of greater than 75% which are otherwise known as high yield pulps.

BACKGROUND OF THE INVENTION

10 In a conventional peroxide bleaching process, sodium hydroxide is used as an alkali source. To achieve a desired brightness with maximum efficiency, auxiliary substances are also used. Such auxiliary substances include sodium silicate, magnesium sulphate and chelating agents inclusive of DTPA (sodium salt of diethylene triamino
15 pentaacetic acid).

Reference may be made to a prior art article by Soteland *et al.*, 1988, TAPPI Proceedings 231-236, which describes a peroxide bleaching process which utilises magnesium oxide as a sole alkaline source. The pulp was pretreated with DTPA and magnesium oxide
20 particles were utilised in a size range of 1.00 mm - 0.25 mm or smaller. The magnesium oxide were also used in a concentration of 2-3% based on the dry weight of the pulp. The MgO used in the process was light-burnt MgO and finely crushed. It was found that brightness levels obtained were very close to that which was achieved
25 by conventional bleaching using NaOH. In the bleaching process, the pulp was diluted to form a pulp suspension and the amount of MgO was added to the suspension under vigorous stirring. Hydrogen peroxide was subsequently added to the suspension at a concentration of 3% based on the weight of the pulp. This reference
30 also made the observation that coarse particles are less effective as an alkaline source during peroxide bleaching.

Having regard to the abovementioned reference, an

observation was also made in the corresponding patent specification DE3617942 that use of MgO as sole alkaline source considerably simplified the bleaching process since sodium hydroxide as alkaline source and auxiliary chemicals such as sodium silicate could be omitted.

Another advantage of using MgO as sole alkaline source was that only a small amount of waste is produced in the bleaching plant. Thus, for example, in integrated mills which produce magnesium sulphite pulp and peroxide bleached high-yield pulps, the used bleaching liquor is combusted and the MgO may be recovered for re-use.

However, the use of MgO as sole alkaline source in a peroxide bleaching process has not as yet achieved widespread commercial acceptance because although the principle of utilising MgO as sole alkaline source was described in the Soteland *et al.* references, the means of reducing the principle to practice on a commercial scale has not yet been fully elucidated.

SUMMARY OF THE INVENTION

Surprisingly, it has now been discovered that commercial usage of magnesium oxide as a sole alkaline source in peroxide bleaching of wood pulp may be achieved by employing MgO particles with a particle size of less than 500 micron and more preferably less than 75 micron and having particle surface area (PSA) of between 20-60 m²/g and more preferably between 30-50 m²/g. By using such parameters, an efficient peroxide bleaching process may be carried out most efficiently on a commercial scale which may be achieved within a maximum bleaching time of 180 minutes and achievement of a maximum target of ISO brightness of 65 in regard to freshly prepared pulp.

Utilizing MgO with parameters outside those stated above will result in a less efficient bleaching process leading to higher usage of chemicals and therefore higher operating costs.

The dosages of MgO that may be utilised in the process of the invention is 0.3-2% based on the weight of the pulp.

The amount of hydrogen peroxide that may be utilised in the process of the invention is from 1-5% based on the weight of the pulp.

To achieve maximum efficiency, the MgO particles must be added to the pulp in the form of a powder or slurry prepared *in situ*.

Preferably the MgO is added to the pulp simultaneously with the peroxide or prior to the addition of the peroxide.

Chelating agents also may be used in the process of the invention and such chelating agents may comprise DTPA, EDTA or HEDTA (hydroxy-ethylene diamine tetracetic acid). Preferably the chelating agent is added to the pulp simultaneously with addition of MgO particles, as well as prior to addition of MgO particles.

Bleaching times of 60-180 minutes may also be utilised by the process of the invention to achieve a target ISO brightness of 55-65.

BRIEF DESCRIPTION OF DRAWINGS

In several preferred embodiments concerning the process of the invention which are discussed hereinafter in relation to Experiments 1 and 2:-

FIG. 1 is a graph showing the effect of particle size on CCS (Cold Caustic Soda) pulp and more specifically showing particle size vs brightness at different times;

FIG. 2 refers to the results of Experiment 2 whereby various samples are plotted against final brightness;

FIG. 3 also refers to the result of Experiment 2 and shows the effect of surface area on CCS pulp and more specifically showing particle size vs brightness at different times; and

FIG. 4 shows the results of FIG. 3 when plotted against time.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

EXPERIMENT 1

EFFECT OF MgO PARTICLE SIZE ON THE BLEACHABILITY OF CCS PULP

5 Introduction

This work was performed to establish a relationship between MgO particle size and alkali performance in the bleaching of CCS (Cold Caustic Soda) wood pulp. Four grades of MgO were trialled, each one identified by its particle size distribution. Each sample of MgO had approximately the same surface area. Particle size and surface area for each of the samples is given in Table 1.

Summary of bleaching work

CCS (chemi-mechanical pulp), pre-treated with DTPA to remove metal ions, was retrieved from the washers in the bleach plant at the Boyer mill. An equivalent mass of 20 grams OD of pulp was weighed out and placed in a plastic breaker. DTPA was then added as 0.15% v/w on the pulp and mixed. MgO as 0.4% w/w, enough water to give a stock consistency of 12% and peroxide as 1.6% v/w on pulp was added and mixed for 2 minutes. The pulp was wrapped in plastic bags and placed into a constant temperature water bath at 65°C. A 3 gram OD sample was removed from the bath at intervals of 2, 3 and 4 hours. This was then made into a brightness hand sheet using the standard Boyer pulp mill method. These were dried overnight in a constant temperature/humidity room and tested for ISO brightness. This procedure was repeated for all MgO samples as well as with control pulp containing no MgO (sample J)

Results

The results of this study indicate that particle size is a key parameter for achieving efficient peroxide bleaching of chemi-mechanical pulp. The results shown in Table 2 and FIG. 1 of this study indicate that an MgO particle size of $< 75 \mu$ (samples G and F, $d_{90} = 65$ and 35 respectively) is required to achieve a target

brightness for a given retention time of 2, 3 or 4 hours.

To achieve an equivalent brightness with samples C ($d_{90} = 1500$) or D ($d_{90} = 3500$), the chemical dosages of MgO and H_2O_2 would need to be increased.

5

EXPERIMENT 2

EFFECT OF MgO PARTICLE SURFACE AREA ON THE BLEACHABILITY OF CCS PULP

Introduction

10 This work was performed to establish a relationship between MgO particle surface area and alkali performance in the bleaching of CCS (Cold Caustic Soda) wood pulp. Five grades of MgO were trialled, each one identifiable by its particle surface area. Each sample of MgO had approximately the same particle size. Particle size and surface area data for each of the samples is given in Table 3.

15 Summary of bleaching work

CCS (chemi-mechanical) pulp, pre-treated with DTPA to remove metal ions, was retrieved from the washers in the bleach plant at the Boyer mill. For each sample, a mass of 10 g O.D. pulp was placed into a beaker and the approximate mass of chemicals added.
20 The pulp was mixed for 2 minutes in a bench top mixer. The pulp was then wrapped in plastic bags and placed into a constant temperature water bath at $65^\circ C$. After two hours retention, the samples were removed from the bath and divided into two. Half the sample was returned to the bath for a further hour of reaction while the other half
25 was made into 5 gram brightness hand sheets. These were dried overnight and then tested for ISO brightness. The work was repeated with samples taken at 2, 3 and 4 hours.

Results

In the previous study (Experiment 1), we determined that
30 MgO particle size was important for peroxide bleaching efficiency. The results of this study indicate that particle surface area is also a key parameter for achieving maximum brightness for a given chemical

dose. The results from these two independent studies (Tables 4 and 5, FIGS. 2 and 3) indicate that a surface area in the range 30-50 m²/g (samples B and C) is required to achieve maximum brightness for a given retention time and chemical dose. Surprisingly, when the surface area is either decreased or increased, the peroxide bleaching efficiency is reduced as indicated in FIGS. 2 and 3 by the bell shaped curves with brightness plateaus between samples B and C. To achieve an equivalent brightness to samples B and C with samples A, D or E, the chemical charges of H₂O and MgO would need to be increased.

The results in FIG. 3, when plotted against time (FIG. 4), appear to indicate that a similar brightness will be achieved with four of the five samples when the bleaching time is extended indefinitely. However, indefinite bleaching time is not a commercial reality and there is a clear benefit, based on these results, in employing MgO particles with a specific size and surface area. In fact, if MgO particles, with parameters outside those stated in this document are used, then the target brightness may not be achieved without increasing chemical dose rates.

TABLES

TABLE 1

Sample	Particle size d90 micron	Surface area m ² /g
F	35	38
G	65	35
H	1500	30
I	3500	30

TABLE 2

Sample	Surface Area m ² /g	Brightness		
		2 hr	3 hr	4 hr
F	< 40	61.39	62.17	62.88
G	< 75	61.22	61.94	62.69
H	< 2000	56.85	57.98	59.17
I	< 5000	56.32	56.19	56.97
J	0	54.3	55.03	55.04

TABLE 3

Sample	Particle size d90 micron	Surface area m ² /g
A	14	1
B	10	35
C	10	43
D	15 (d90 = 70)	98
E	11	142

TABLE 4

Sample	Surface Area m ² /g	Brightness	
		2 hr	3 hr
A	1	58.5	59.1
B	35	60.2	60.5
C	43	60.1	60.6
D	98	58.3	59.0
E	142	56.8	58.8

TABLE 5

Sample	Surface Area m ² /g	Brightness		
		2 hr	3 hr	4 hr
A	1	56.02	57.75	58.54
B	35	58.89	60.58	60.96
C	43	59.17	31.37	61.09
D	98	58.15	59.1	60.29
E	142	57.45	59.13	60.27

LEGENDS**TABLE 2**Bleaching conditions:

MgO % w/w on oven dry pulp = 0.4%

5 H₂O₂ - 1.6%

DTPA = 0.15%

Temperature = 65°C

Initial brightness 47.1

TABLE 4

10 Bleaching conditions:

MgO % w/w on oven dry pulp = 0.3%

H₂O₂ = 1.8%

DTPA = 0.1%

Temperature = 65°C

15 Initial brightness 43.5

TABLE 5Bleaching conditions:

MgO % w/w on oven dry pulp = 0.4%

H₂O₂ = 1.6%

20 DTPA = 0.15%

Temperature - 65°C

Initial brightness 47.1

FIG. 1

25 Effect of particle size on CCS pulp - Particle size vs brightness at
different times.

FIG. 2

Sample number vs final final brightness.

FIG. 3

30 Effect of surface area on CCS pulp - Surface area vs brightness at
different

FIG. 4

Effect of surface area on CCS pulp - Time vs brightness for different surface areas

CLAIMS

1. A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g.
2. A process as claimed in Claim 1 wherein said MgO particles have a particle size of less than 75 microns.
3. A process as claimed in Claim 1 wherein the MgO particles have a particle size of between 30-50 m²/g.
4. A process as claimed in Claim 1 which utilises a dosage of MgO which is 0.3-2% based on the weight of the pulp.
5. A process as claimed in Claim 1 wherein the amount of hydrogen peroxide utilised comprises 1-5% based on the weight of the pulp.
6. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp in the form of a powder or slurry prepared *in situ*.
7. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp simultaneously with the addition of peroxide.
8. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp prior to the addition of peroxide.
9. A process as claimed in Claim 1 wherein a chelating agent is added to the pulp selected from DTPA, EDTA or HEDTA.
10. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp simultaneously with the addition of MgO particles.
11. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp prior to the addition of MgO particles.

AMENDED CLAIMS

[received by the International Bureau on 3 October 1996 (3.10.96); original claims 1 and 3 amended; remaining claims unchanged (1 page)]

1. A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a maximum target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g.
2. A process as claimed in Claim 1 wherein said MgO particles have a particle size of less than 75 microns.
3. A process as claimed in Claim 1 wherein the MgO particles have a particle surface area of between 30-50 m²/g.
4. A process as claimed in Claim 1 which utilises a dosage of MgO which is 0.3-2% based on the weight of the pulp.
5. A process as claimed in Claim 1 wherein the amount of hydrogen peroxide utilised comprises 1-5% based on the weight of the pulp.
6. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp in the form of a powder or slurry prepared *in situ*.
7. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp simultaneously with the addition of peroxide.
8. A process as claimed in Claim 1 wherein the MgO particles is added to the pulp prior to the addition of peroxide.
9. A process as claimed in Claim 1 wherein a chelating agent is added to the pulp selected from DTPA, EDTA or HEDTA.
10. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp simultaneously with the addition of MgO particles.
11. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp prior to the addition of MgO particles.

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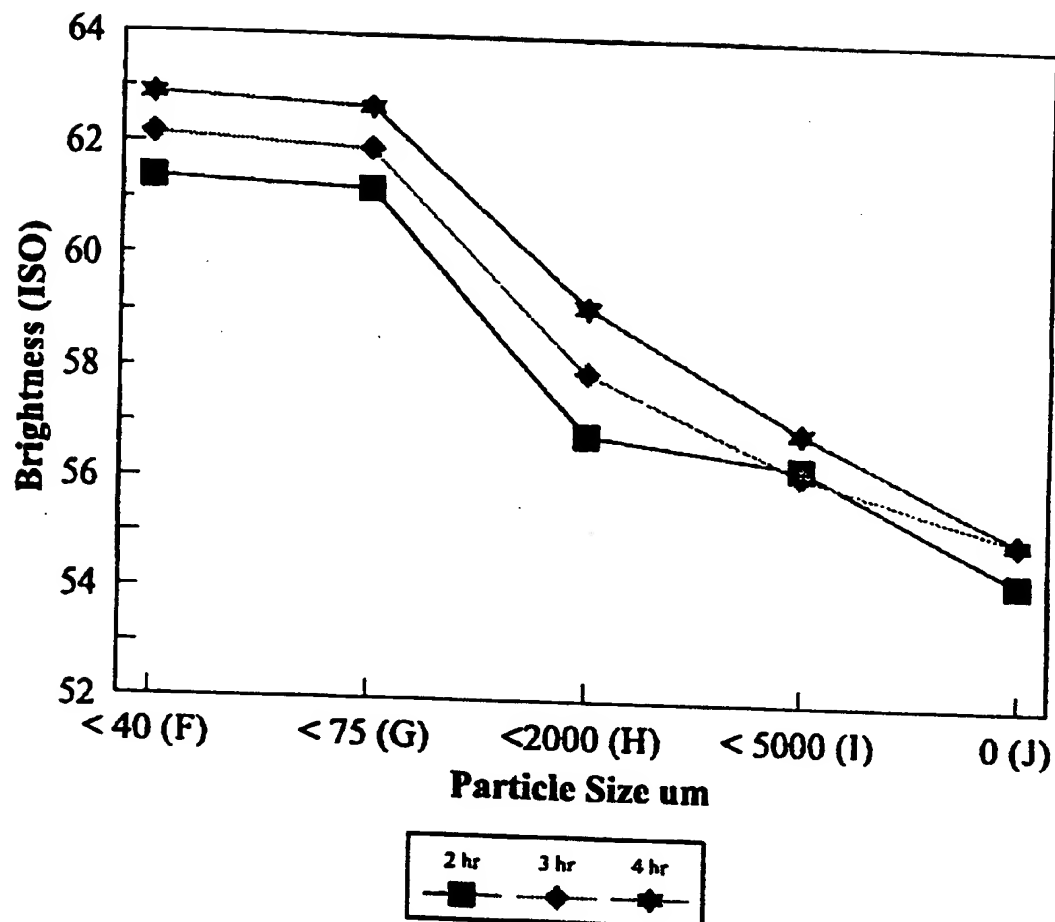


FIG. 1

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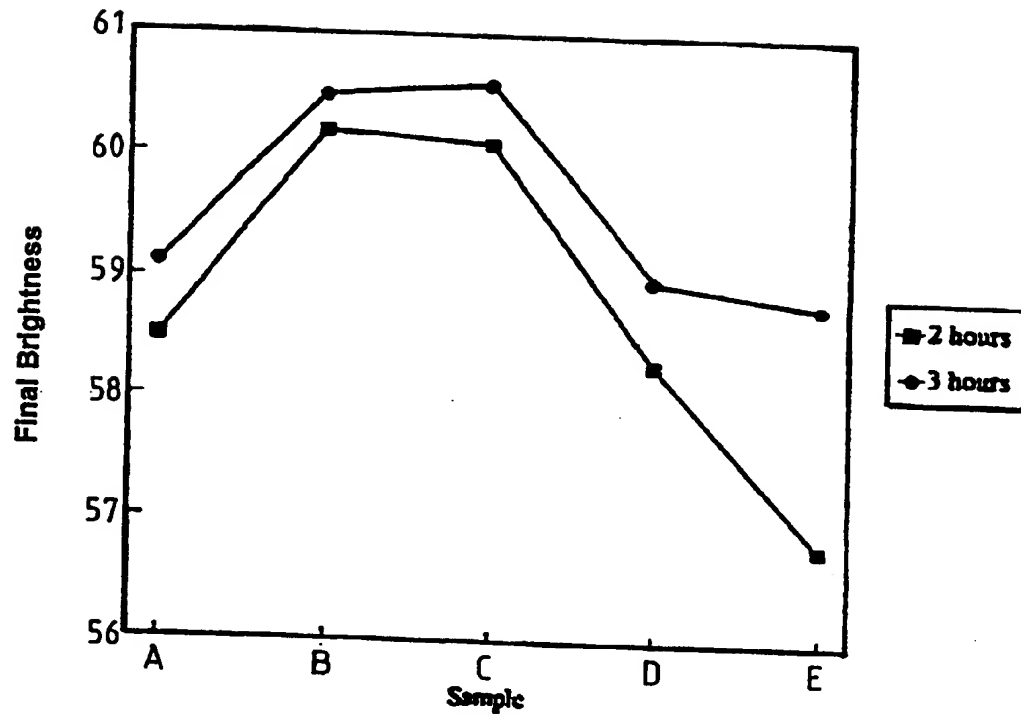


FIG. 2

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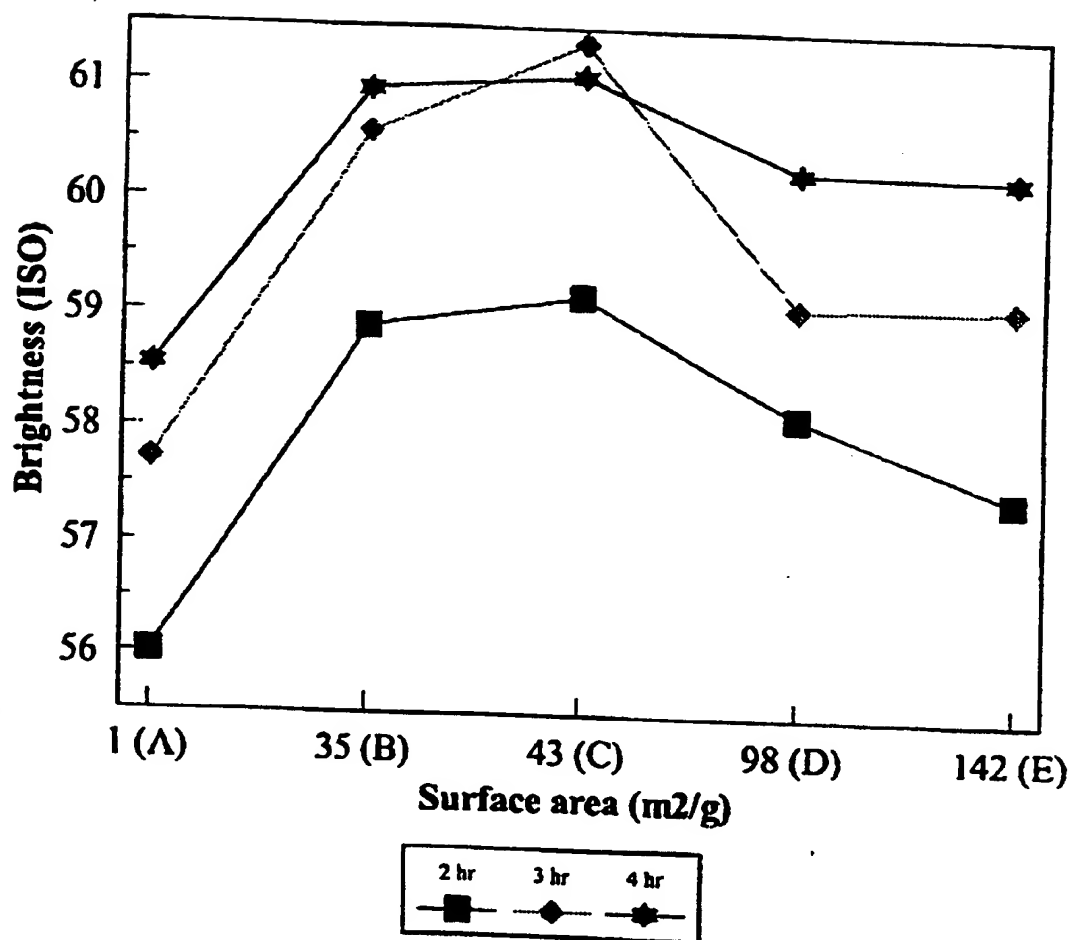


FIG. 3

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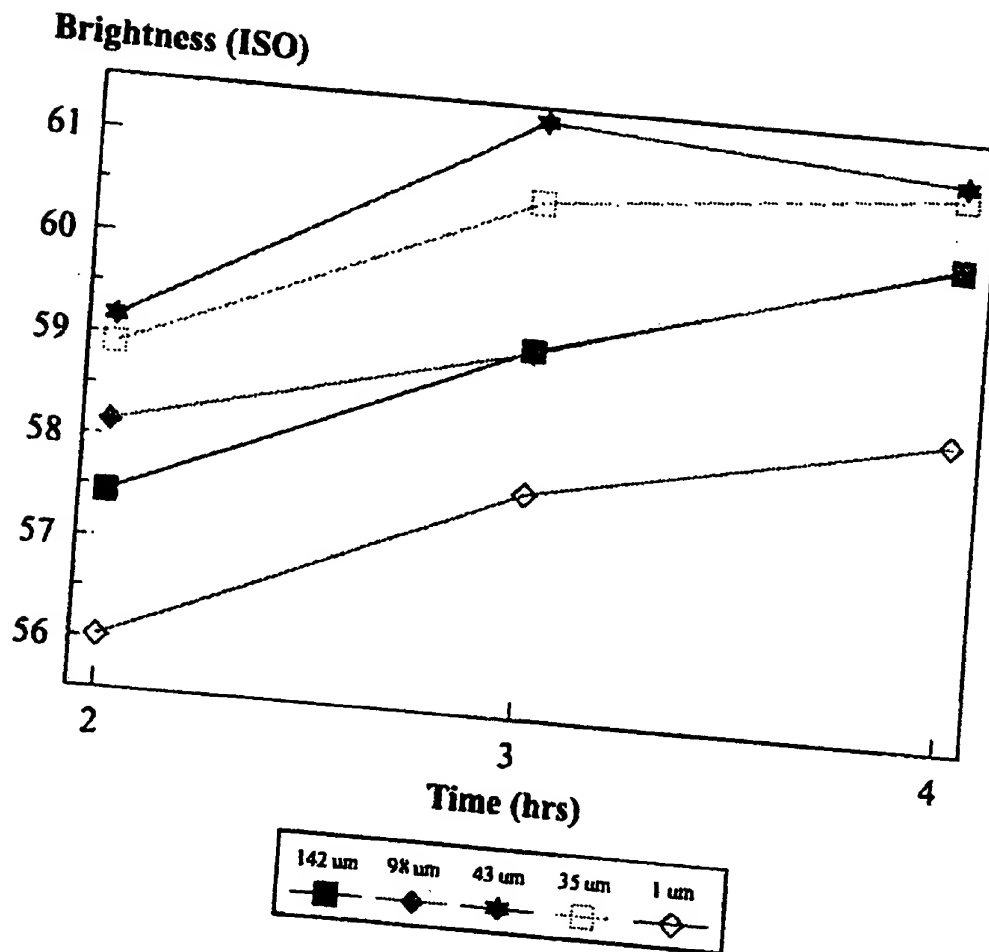



FIG. 4

INTERNATIONAL SEARCH REPORT

International Application No.
PCT/AU 96/00354

A. CLASSIFICATION OF SUBJECT MATTER		
Int Cl ⁶ : D21C 9/16		
According to International Patent Classification (IPC) r to both national classification n and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) D21C 9/16		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched AU: IPC as above		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) DERWENT		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY PROCEEDINGS 1988, pages 231 - 236, N. SOTELAND, "Use of MgO or CaO as the only alkaline source in peroxide bleaching of high yield pulps", Whole document.	1 -6
X,P	DE 4400954 A (KUERZEDER S) 10 August 1995, See col. 3 lines 56 - 64, claim 1.	1 - 6, 9
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C <input checked="" type="checkbox"/> See patent family annex		
<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&" document member of the same patent family</p>		
Date of the actual completion of the international search 22 August 1996		Date of mailing of the international search report 30 AUG 1996
Name and mailing address of the ISA/AU AUSTRALIAN INDUSTRIAL PROPERTY ORGANISATION PO BOX 200 WODEN ACT 2606 AUSTRALIA Facsimile No.: (06) 285 3929		Authorized officer  S. DEUIS Telephone No.: (06) 283 2146

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/AU 96/00354

C (Continuation)

DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 3617942 A (PAPIRIND FORSKNINGS) 11 December 1986.	
A	US 3951732 A (SJOSTROM et al.) 20 April 1976.	

INTERNATIONAL SEARCH REPORT
Information on patent family members

International Application No.
PCT/AU 96/00354

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
DE	3617942	FI	862437	FR	2583078	NO	852330
		SE	8602065				
US	3951732	CA	1026908	DE	2355741	ES	420507
		FI	52877	FR	2207215	JP	49133601
		JP	51012723	NO	137015		

END OF ANNEX

PEROXIDE BLEACHING OF PULP

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Publication date: 1996-12-27
Inventor(s): VINCENT ADAM HAYDEN (AU); MCLEAN IAN ALEXANDER (AU)
Applicant(s): ICI AUSTRALIA OPERATIONS (AU); QUEENSLAND METALS CORP LTD (AU); VINCENT ADAM HAYDEN (AU); MCLEAN IAN ALEXANDER (AU)
Requested Patent: ☐ WO9641917
Application Number: WO1996AU00354 19960613
Priority Number (s): AU1995PN03520 19950613
IPC Classification: D21C9/16
EC Classification: D21C9/10F4, D21C9/16B
Equivalents: CA2224677, DE69611091D, ☐ EP0832323 (WO9641917), A4, B1
Cited Documents: DE4400954; DE3617942; US3951732

Abstract

A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g. By using such parameters, a peroxide bleaching process may be carried out most efficiently on a commercial scale.

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Description

TITLE

"PEROXIDE BLEACHING OF PULP"

FIELD OF THE INVENTION

THIS INVENTION relates to a process for peroxide bleaching of pulp. Pulp which may be bleached in the process of the invention include lignocellulose pulp which may be produced mechanically and chemi-mechanically with yields in the region of greater than 75% which are otherwise known as high yield pulps.

BACKGROUND OF THE INVENTION

In a conventional peroxide bleaching process, sodium hydroxide is used as an alkali source. To achieve a desired brightness with maximum efficiency, auxiliary substances are also used. Such auxiliary substances include sodium silicate, magnesium sulphate and chelating agents inclusive of DTPA (sodium salt of diethylene triamino pentaacetic acid).

Reference may be made to a prior art article by Soteland et al., 1988, TAPPI Proceedings 231-236, which describes a peroxide bleaching process which utilises magnesium oxide as a sole alkaline source. The pulp was pretreated with DTPA and magnesium oxide particles were utilised in a size range of 1.00 mm - 0.25 mm or smaller. The magnesium oxide were also used in a concentration of 23% based on the dry weight of the pulp. The MgO used in the process was light-burnt MgO and finely crushed. It was found that brightness levels obtained were very close to that which was achieved by conventional bleaching using NaOH. In the bleaching process, the pulp was diluted to form a pulp suspension and the amount of MgO was added to the suspension under vigorous stirring. Hydrogen peroxide was subsequently added to the suspension at a concentration of 3% based on the weight of the pulp. This reference also made the observation that coarse particles are less effective as an alkaline source during peroxide bleaching.

Having regard to the abovementioned reference, an observation was also made in the corresponding patent specification

DE3617942 that use of MgO as sole alkaline source considerably simplified the bleaching process since sodium hydroxide as alkaline source and auxiliary chemicals such as sodium silicate could be omitted.

Another advantage of using MgO as sole alkaline source was that only a small amount of waste is produced in the bleaching plant. Thus, for example, in integrated mills which produce magnesium sulphite pulp and peroxide bleached high-yield pulps, the used bleaching liquor is combusted and the MgO may be recovered for re-use.

However, the use of MgO as sole alkaline source in a peroxide bleaching process has not as yet achieved widespread commercial acceptance because although the principle of utilising

MgO as sole alkaline source was described in the Soteland et al.

references, the means of reducing the principle to practice on a commercial scale has not yet been fully elucidated.

SUMMARY OF THE INVENTION

Surprisingly, it has now been discovered that commercial usage of magnesium oxide as a sole alkaline source in peroxide bleaching of wood pulp may be achieved by employing MgO particles with a particle size of less than 500 micron and more preferably less than 75 micron and having particle surface area (PSA) of between 2060 m²/g and more preferably

between 30-50 m²/g. By using such parameters, an efficient peroxide bleaching process may be carried out most efficiently on a commercial scale which may be achieved within a maximum bleaching time of 180 minutes and achievement of a maximum target of ISO brightness of 65 in regard to freshly prepared pulp.

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Preferably the MgO is added to the pulp simultaneously with the peroxide or prior to the addition of the peroxide.

Chelating agents also may be used in the process of the invention and such chelating agents may comprise DTPA, EDTA or HEDTA (hydroxy-ethylene diamine tetracetic acid). Preferably the chelating agent is added to the pulp simultaneously with addition of MgO particles, as well as prior to addition of MgO particles.

Bleaching times of 60-180 minutes may also be utilised by the process of the invention to achieve a target ISO brightness of 55-65.

BRIEF DESCRIPTION OF DRAWINGS

In several preferred embodiments concerning the process of the invention which are discussed hereinafter in relation to

Experiments 1 and 2:

FIG. 1 is a graph showing the effect of particle size on CCS (Cold Caustic Soda) pulp and more specifically showing particle size vs brightness at different times;

FIG. 2 refers to the results of Experiment 2 whereby various samples are plotted against final brightness;

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DESCRIPTION OF THE PREFERRED EMBODIMENTS

EXPERIMENT 1

EFFECT OF MGO PARTICLE SIZE ON THE BLEACHABILITY OF CCS PULP

Introduction

This work was performed to establish a relationship between MgO particle size and alkali performance in the bleaching of

CCS (Cold Caustic Soda) wood pulp. Four grades of MgO were trialled, each one identified by its particle size distribution. Each sample of MgO had approximately the same surface area. Particle size and surface area for each of the samples is given in Table 1.

Summary of bleaching work

CCS (chemi-mechanical pulp), pre-treated with DTPA to remove metal ions, was retrieved

from the washers in the bleach plant at the Boyer mill. An equivalent mass of 20 grams OD of pulp was weighed out and placed in a plastic breaker. DTPA was then added as 0.15% v/w on the pulp and mixed. MgO as 0.4% w/w, enough water to give a stock consistency of 12% and peroxide as 1.6% v/w on pulp was added and mixed for 2 minutes. The pulp was wrapped in plastic bags and placed into a constant temperature water bath at 65°C. A 3 gram OD sample was removed from the bath at intervals of 2, 3 and 4 hours. This was then made into a brightness hand sheet using the standard Boyer pulp mill method. These were dried overnight in a constant temperature/humidity room and tested for ISO brightness. This procedure was repeated for all MgO samples as well as with control pulp containing no MgO (sample J)

Results

The results of this study indicate that particle size is a key parameter for achieving efficient peroxide bleaching of chemimechanical pulp. The results shown in Table 2 and FIG. 1 of this study indicate that an MgO particle size of $< 75\ \mu\text{m}$ (samples G and F, $d_{90} = 65$ and 35 respectively) is required to achieve a target brightness for a given retention time of 2, 3 or 4 hours.

To achieve an equivalent brightness with samples C ($d_{90} = 1500$) or D ($d_{90} = 3500$), the chemical dosages of MgO and H₂O₂ would need to be increased.

EXPERIMENT 2

EFFECT OF MgO PARTICLE SURFACE AREA ON THE BLEACHABILITY OF CCS PULP

Introduction

This work was performed to establish a relationship between MgO particle surface area and alkali performance in the bleaching of CCS (Cold Caustic Soda) wood pulp. Five grades of MgO were trialled, each one identifiable by its particle surface area. Each sample of MgO had approximately the same particle size. Particle size and surface area data for each of the samples is given in Table 3.

Summary of bleaching work

CCS (chemi-mechanical) pulp, pre-treated with DTPA to remove metal ions, was retrieved from the washers in the bleach plant at the Boyer mill. For each sample, a mass of 10 g O.D. pulp was placed into a beaker and the approximate mass of chemicals added.

The pulp was mixed for 2 minutes in a bench top mixer. The pulp was then wrapped in plastic bags and placed into a constant temperature water bath at 65°C. After two hours retention, the samples were removed from the bath and divided into two. Half the sample was returned to the bath for a further hour of reaction while the other half was made into 5 gram brightness hand sheets. These were dried overnight and then tested for ISO brightness. The work was repeated with samples taken at 2, 3 and 4 hours.

Results

In the previous study (Experiment 1), we determined that MgO particle size was important for peroxide bleaching efficiency. The results of this study indicate that particle surface area is also a key parameter for achieving maximum brightness for a given chemical dose. The results from these two independent studies (Tables 4 and 5, FIGS. 2 and 3) indicate that a surface area in the range 30-50 m²/g (samples B and C) is required to achieve maximum brightness for a given retention time and chemical dose. Surprisingly, when the surface area is either decreased or increased, the peroxide bleaching efficiency is reduced as indicated in FIGS. 2 and 3 by the bell shaped curves with brightness plateaus between samples B and C. To achieve an equivalent brightness to samples B and C with samples A, D or E, the chemical charges of H₂O and MgO would need to be increased.

The results in FIG. 3, when plotted against time (FIG. 4), appear to indicate that a similar brightness will be achieved with four of the five samples when the bleaching time is extended indefinitely.

However, indefinite bleaching time is not a commercial reality and there is a clear benefit, based on these results, in employing MgO particles with a specific size and surface area. In fact, if MgO particles, with parameters outside those stated in this document are used, then the target brightness may not be achieved without increasing chemical dose rates.

TABLES

TABLE 1

Sample	Particle size d90 micron	Surface area m ² /g
F	35	38
G	65	35
H	1500	30
I	3500	30

TABLE 2

Sample	Surface Area m ² /g	Brightness 2 hr	Brightness 3 hr	Brightness 4 hr
F	< 40	61.39	62.17	62.88
G	< 75	61.22	61.94	62.69
H	< 2000	56.85	57.98	59.17
I	< 5000	56.32	56.19	56.97
J	0	54.3	55.03	55.04

TABLE 3

Sample	Particle size d90 micron	Surface area m/g
A	14	1
B	10	35
C	10	43
D	15 (d90 = 70)	98
E	1	11 142

TABLE 4

Sample	Surface Area m/g	Brightness 2 hr	Brightness 3 hr
A	1	58.5	59.1
B	35	60.2	60.5
C	43	60.1	60.6
D	98	58.3	59.0
E	142	56.8	58.8

TABLE 5

Sample Surface Area Brightness

m/g 2 hr 3 hr 4 hr

A 1 56.02 57.75 58.54

B 35 58.89 60.58 60.96

C 43 59.17 31.37 61.09

D 98 58.15 59.1 60.29

E 142 57.45 59.13 60.27

LEGENDS

TABLE 2

Bleaching conditions:

MgO % w/w on oven dry pulp = 0.4% H₂O₂ = 1.6%

DTPA = 0.15%

Temperature = 650C

Initial brightness 47.1

TABLE 4

Bleaching conditions:

MgO % w/w on oven dry pulp = 0.3% H₂O₂ = 1.8%

DTPA = 0.1%

Temperature = 650C

Initial brightness 43.5

TABLE 5

Bleaching conditions:

MgO % w/w on oven dry pulp = 0.4% H₂O₂ = 1.6%

DTPA = 0.15%

Temperature = 65 OC

Initial brightness 47.1

FIG. 1

Effect of particle size on CCS pulp - Particle size vs brightness at different times.

FIG. 2

Sample number vs final final brightness.

FIG. 3

Effect of surface area on CCS pulp - Surface area vs brightness at different

FIG. 4

Effect of surface area on CCS pulp - Time vs brightness for different surface areas

CLAIMS 1. A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g.

2. A process as claimed in Claim 1 wherein said MgO particles have a particle size of less than 75 microns.

3. A process as claimed in Claim 1 wherein the MgO particles have a particle size of between 30-50 m²/g.

4. A process as claimed in Claim 1 which utilises a dosage of MgO which is 0.3-2% based on the weight of the pulp.

5. A process as claimed in Claim 1 wherein the amount of hydrogen peroxide utilised comprises 1-5% based on the weight of the pulp.

6. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp in the form of a powder or slurry prepared in situ.

7. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp simultaneously with the addition of peroxide.

8. A process as claimed in Claim 1 wherein the MgO particles is added to the pulp prior to the addition of peroxide.

9. A process as claimed in Claim 1 wherein a chelating agent is added to the pulp selected from DTPA, EDTA or HEDTA.

10. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp simultaneously with the addition of MgO particles.

11. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp prior to the addition of MgO particles.

AMENDED CLAIMS

[received by the International Bureau on 3 October 1996 (3.10.96); original claims 1 and 3 amended; remaining claims unchanged (1 page)]

1. A process for peroxide bleaching of pulp using magnesium oxide as sole alkaline source wherein said pulp is bleached in the presence of hydrogen peroxide for a maximum period of 180 minutes and achievement of a maximum target ISO brightness of 65 in regard to freshly prepared pulp characterised in that said magnesium oxide is utilised as MgO particles having a particle size of 5-500 microns and a particle surface area (PSA) of between 20-60 m²/g.

2. A process as claimed in Claim 1 wherein said MgO particles have a particle size of less than 75 microns.

3. A process as claimed in Claim 1 wherein the MgO particles have a particle surface area of between 30-50 m²/g.

4. A process as claimed in Claim 1 which utilises a dosage of MgO which is 0.3-2% based on the weight of the pulp.

5. A process as claimed in Claim 1 wherein the amount of hydrogen peroxide utilised comprises 1-5% based on the weight of the pulp.

6. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp in the form of a powder or slurry prepared in situ.

7. A process as claimed in Claim 1 wherein the MgO particles are added to the pulp simultaneously with the addition of peroxide.

8. A process as claimed in Claim 1 wherein the MgO particles is added to the pulp prior to the addition of peroxide.

9. A process as claimed in Claim 1 wherein a chelating agent is added to the pulp selected from DTPA, EDTA or HEDTA.

10. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp simultaneously with the addition of MgO particles.

11. A process as claimed in Claim 9 wherein the chelating agent is added to the pulp prior to the addition of MgO particles

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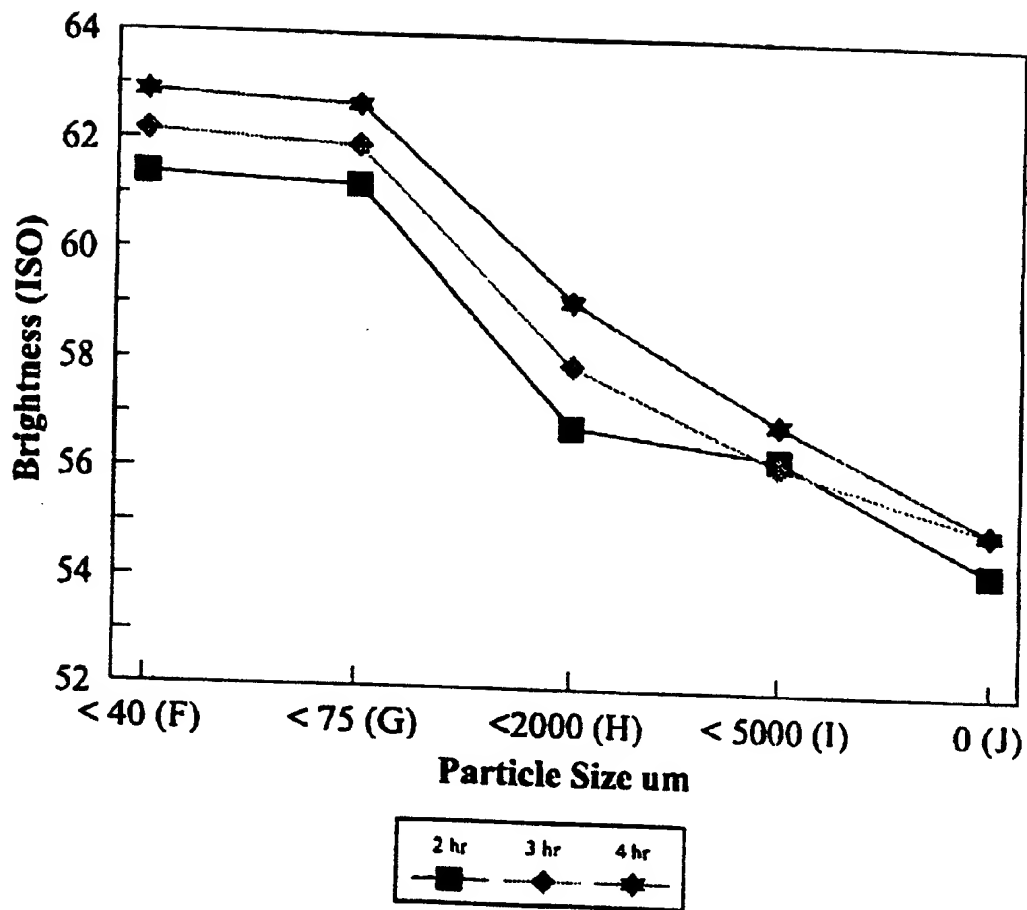


FIG. 1

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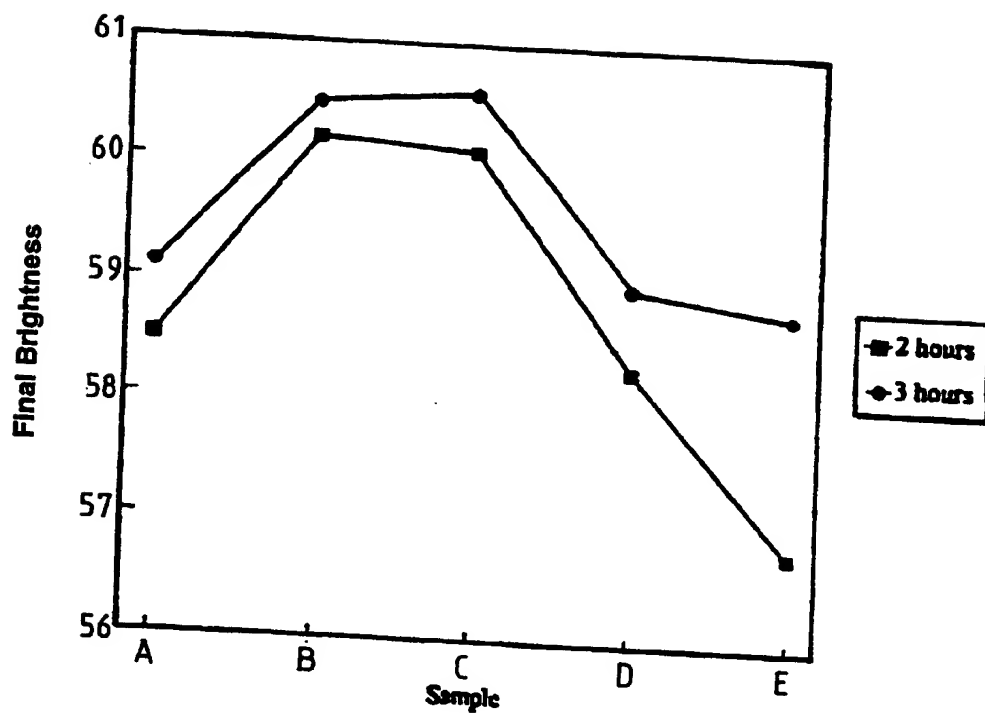


FIG. 2

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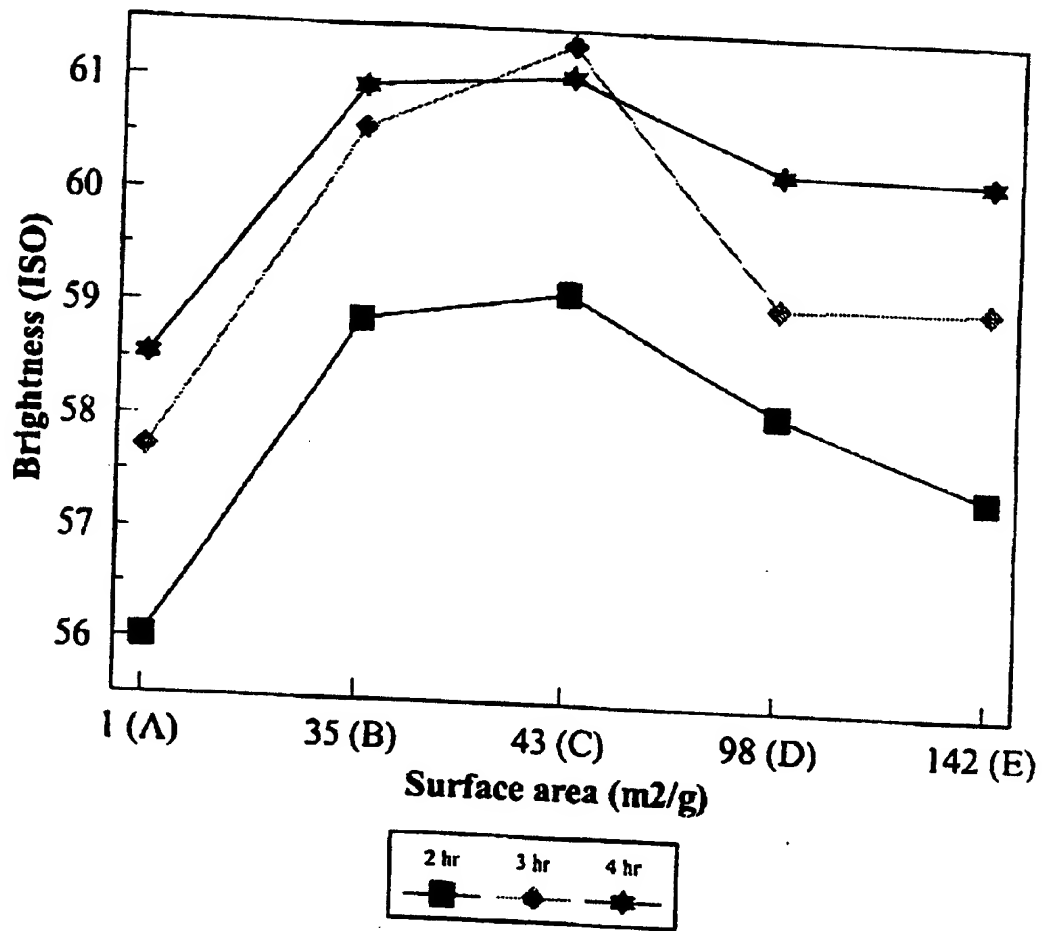


FIG. 3

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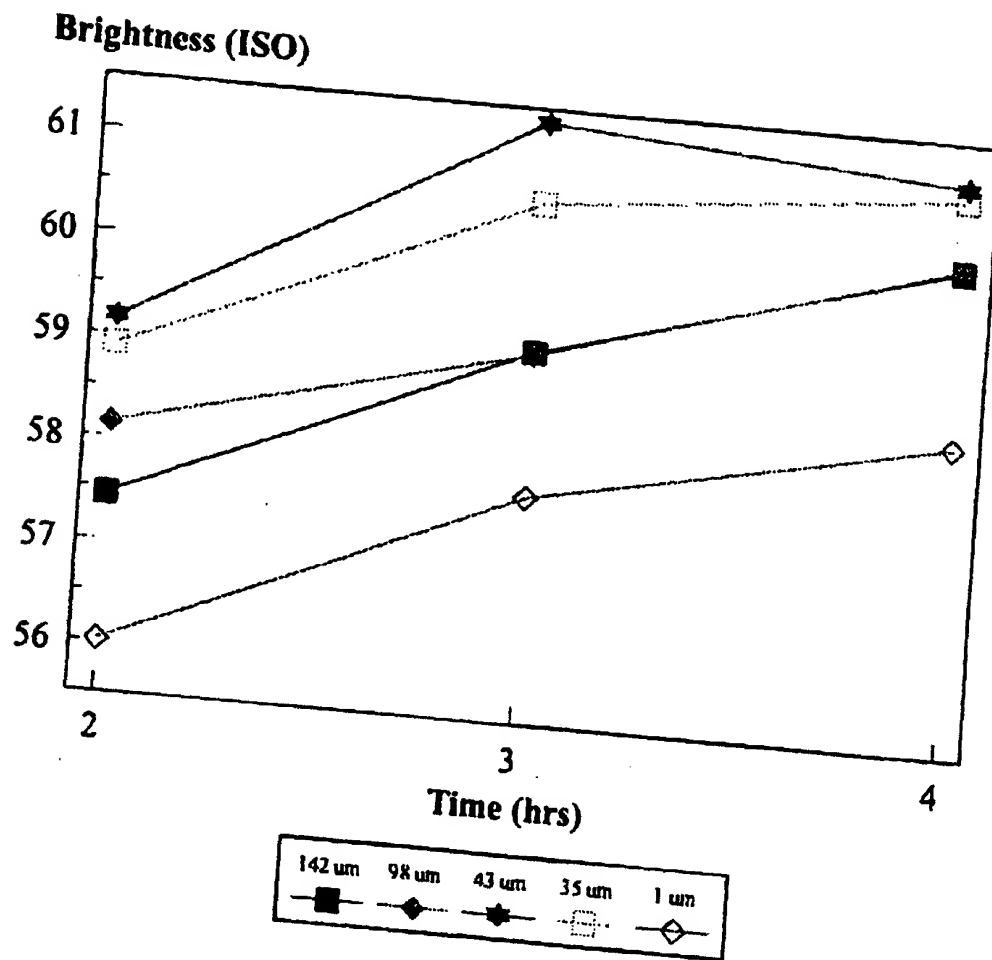


FIG. 4